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ADHESION IN ROCK

George A. Savanick, et al

Bureau of Mines

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13. ABSTRACT

A direct method has been developed for estimating the strength of intergranular adhesion in rock. It involves the separation of a bicrystal from the rock and a determination of the tensile strength at the solid-solid interface. This technique has been successfully applied to the study of quartz-feldspar interfaces separated from pegmatites, graphic granite, and the Rockville granite yielding average tensile strengths of 5.86, 8.62, and 10.65 MN/m^2 (850, 1,250, and 1,524 psi) respectively. The data generated by this technique indicate that the members of these bicrystals are strongly adherent. Examination of bicrystals broken at the crystalline interfaces indicates that the bonds responsible for this adhesion operate only over a portion of the interfacial area.

The significance of this work is that it demonstrates that tensile strength tests can be conducted on small selected areas, e.g. grain boundaries in rock. This permits a determination of small scale zones of strength or weakness which may be related to the overall strength of the rock. Selected area microindentation hardness testing has also been performed to probe hardness variations near phase boundaries.

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FINAL REPORT
ON
ADHESION IN ROCKS

by

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Donald I. Johnson[2]

ABSTRACT

The objective of this research is to ascertain the magnitude of the forces responsible for the coherency of rock by quantifying the strength of the attractive forces operating at intercrystalline interfaces in rock. These forces act in opposition to the stresses set up in various rock fragmentation processes, hence measurements of the strength of these attractive forces might prove useful in the design of more efficient methods of rock fragmentation.

A direct method has been developed for estimating the strength of intergranular adhesion in rock. It involves the separation of a bicrystal from the rock and a determination of the tensile strength at the solid-solid interface. This technique has been successfully applied to the study of quartz-feldsp : interfaces separated from pegmatites, graphic granite, and the Rockville granite yielding average tensile strengths of 5.86, 8.62, and 10.65 MN/m^2 (850, 1,250, and 1,524 psi) respectively. The data generated by this technique indicate that the members of these bicrystals are strongly adherent. Examination of bicrystals broken at the crystalline interfaces indicates that the bonds responsible for this adhesion operate only over a portion of the interfacial area.

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The significance of this work is that it demonstrates that tensile strength tests can be conducted on small selected areas, e.g. grain boundaries in rock. This permits a determination of small scale zones of strength or weakness which may be related to the overall strength of the rock.

Selected area microindentation hardness testing has also been performed to probe hardness variations near phase boundaries.

INTRODUCTION

Rock disaggregation is a process whereby energy is applied to overcome the attractive forces holding the rock together. Characterization of these forces would seem to be a prerequisite for the optimization of this process in order to realize a more efficient expenditure of energy with consequent lowering of cost.

The chemical bonds responsible for the mutual attraction of atoms which gives rise to the cohesion of crystals have long been an object of study (1)[3]. The chemical bond strengths have been measured and are tabulated (1,2). On the other hand, very little effort has been expended in understanding adhesion at phase boundaries in rock, i.e., the mechanism by which the constituent minerals are joined together at crystalline interfaces to form a coherent polycrystalline aggregate. Thus no measurements have been made of the adhesive strength at crystalline interfaces in rock.

The objective of this research is to fill this void by developing a method of selected area tensile strength testing by which the tensile

[3] Underlined numbers in parentheses refer to items in the list of references.

strength of crystalline interfaces could be measured and to present a tabulation and an interpretation of these data.

No previous measurements of the adhesive strength of crystalline interfaces in solids are known to the authors. This may be a reflection of the difficulties inherent in such measurements. The crystalline interface must be separated from the rock prior to any tensile strength testing. This can be very tedious, and in itself, might discourage investigators.

It must also be realized that pure adhesive failure is an idealization which can be approximated but cannot be attained. The requirement that the rupture must occur precisely at the atomic boundary between two phases renders pure adhesive failure very improbable. This is a problem that confronts any destructive testing of adhesive joints in the adhesives industry (3). Bits of one mineral will adhere to the other member of the broken bicrystal. This prohibits a direct measurement of the number of bonds which bind one mineral to the other, but it permits a determination of the areal extent of the bonding between the grains.

The limitations inherent in this type of experimentation tend to narrow the scope of the research. Since adhesive strength is defined as the resistance of an interface to tensile stresses, the crystalline interfaces were subjected only to tensile strength tests. The tests were limited to planar interfaces in an attempt to eliminate the strengthening contribution from macroscopic interlocking of phases. In addition the difficulties of extraction and the errors in the tensile strength measurement increase as the size of the bicrystal decreases. In view of these difficulties, it was decided to limit consideration to relatively large quartz-feldspar

bicrystals extracted from pegmatites, graphic granites, and coarse grained granites.

The effort was focused on quartz-feldspar bicrystals because they are the building blocks of all granitic rocks. Hence it may be possible to relate the adhesive strength data to the strength and mode of fracture of granitic rocks.

This report contains a description of a method for extracting bicrystals from selected areas in granitic rocks and for determining the tensile strength at the crystalline interfaces. Adhesive strength data for crystalline interfaces extracted from pegmatites, graphic granites, and the Rockville granite are tabulated and compared with the tensile strength of quartz and f ldspar taken from the same rock. This permits an assessment of the strength of adhesion at crystalline interfaces and a comparison with the cohesive strength of the constituent minerals.

EXPERIMENTAL PROCEDURES

In adhesion technology, adhesion is defined (4) as the force per unit area required to separate two solids in contact. The magnitude of this stress can only be estimated from the results of destructive testing (5). The most easily interpreted measure of adhesion is the normal tensile force required for separation, hence it was necessary to develop a method of selected area tensile strength testing to measure the adhesion at crystalline interfaces in rocks.

A successful method for selected area tensile strength testing must provide for the extraction of planar intercrystalline boundaries from the rock and permit the separation of the joined crystals at the crystalline interface.

A technique which meets these requirements has been devised and is composed of the following sequence of steps:

(1) Rocks containing crystalline interfaces of interest are cut into thin slices. The thickness of these slices is dictated by the extent of the crystalline interfaces. The pegmatites studied in this research were cut 12 mm (1/2 inch) thick, whereas Rockville granite and graphic granites were cut into 6 mm (1/4 inch) thick slices.

(2) These slabs (fig. 1) were fastened to the surface of a block of soft wood with a fast drying epoxy resin. The wood surface is coated with enamel paint in order to facilitate removal of the rock-epoxy ensemble to permit reuse of the wooden block.

(3) An area containing the trace of a planar crystalline interface is selected and removed by drilling with a diamond core bit mounted in a drill press (fig. 2). It was found that 12 mm (1/2 inch) O.D. core drills were ideal for removing quartz-feldspar boundaries from pegmatites whereas a 5 mm (1/4 inch) O.D. core drill worked well for extracting quartz-feldspar bicrystals from the Rockville granite and graphic granite.

(4) Those portions of the crystalline interface which are non-planar or off center are removed by grinding perpendicular to the cylinder axis with a thin section grinder (fig. 3).

(5) The diameter of the cylinder and the length of the cylinder axis is measured with calipers or a micrometer.

FIGURE 1. - Slabs of Graphic Granite Cemented to a Wooden Block.

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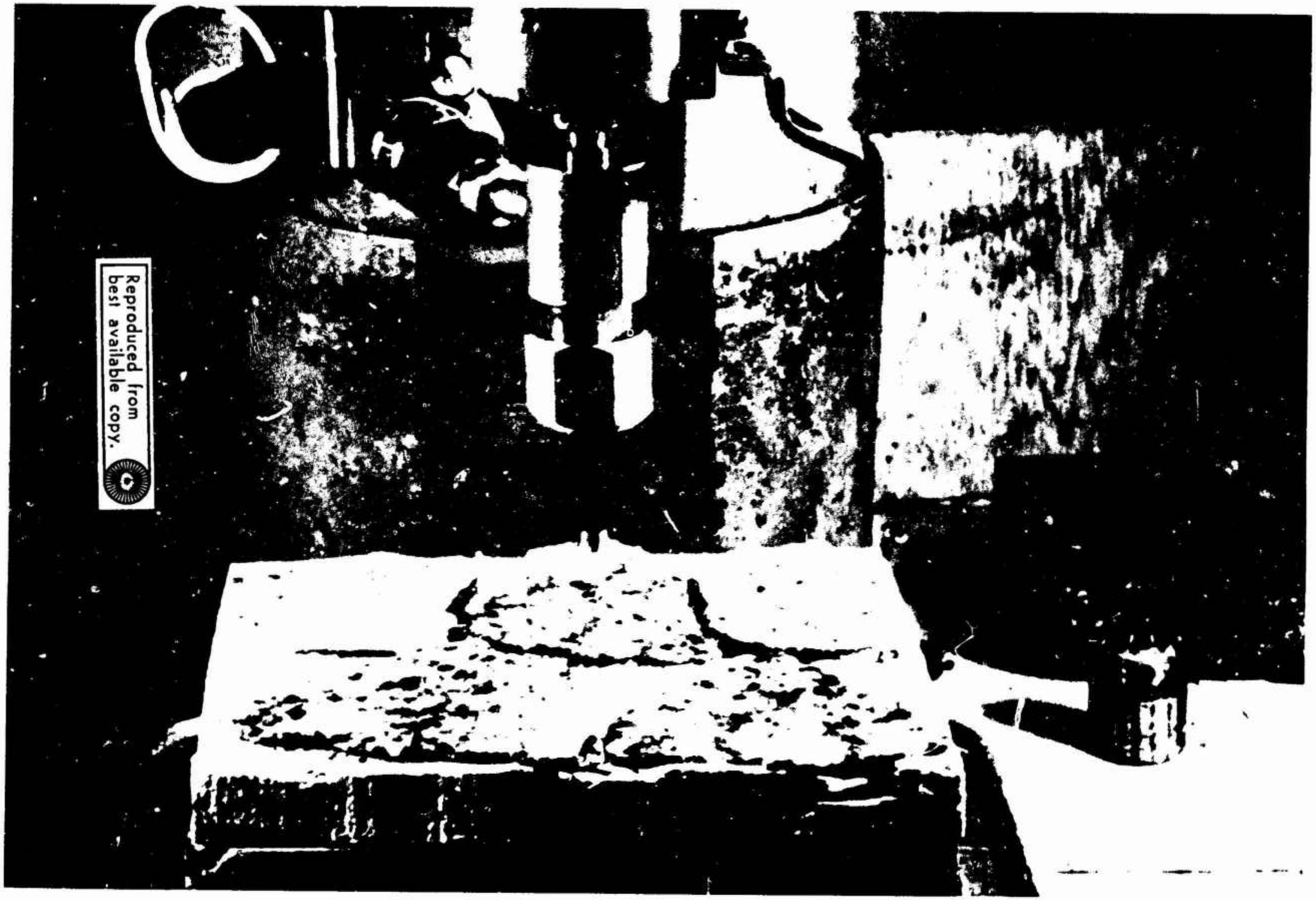

FIGURE 2. - Rock Samples in Place Under a Diamond Tipped Core Drill.

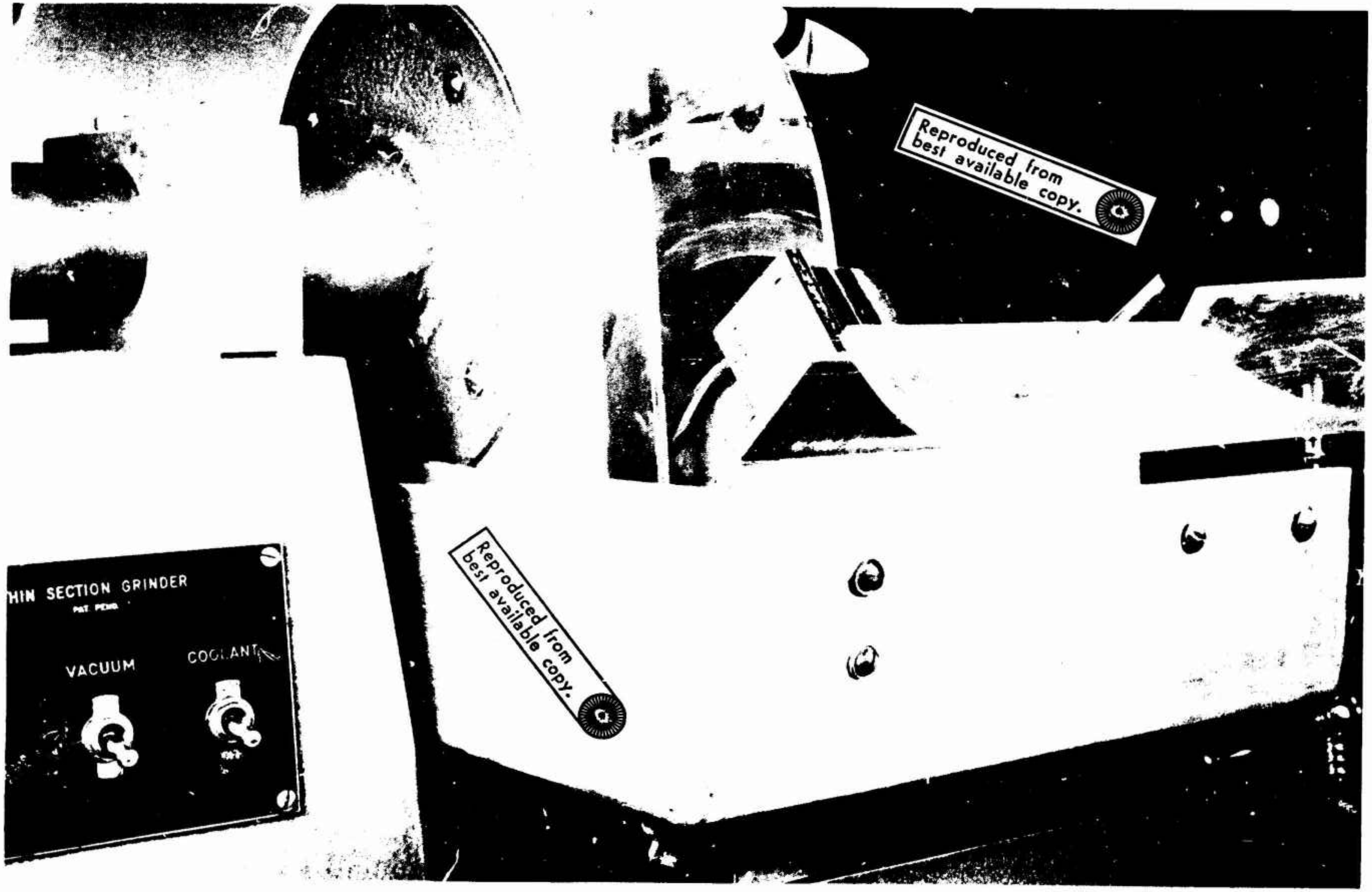

FIGURE 3. - Bicrystal Mounted on the Arm of a Thin Section Grinder.

(6) The sample is placed in an Instron testing machine (fig. 4) and subjected to an indirect tensile (Braxilian) test. The sample is oriented so that the stress is concentrated and the sample breaks at the crystalline interface.

(7) The tensile strength of the intercrystalline boundary is calculated using the formula:

$$T.\ S. = \frac{2L}{\pi d l}$$

Where L is the load applied to the sample, d is the diameter of the sample, and l is the length of the cylinder axis.

This technique results in a bicrystal sample (fig. 5) that has separated at the crystalline boundary and yields a quantitative measure of the intergranular adhesive strength.

EXPERIMENTAL DATA

The data generated by the technique described above are tabulated in this section. The tables are organized not only to illustrate the magnitude of the adhesive strength at the crystalline interfaces, but also to permit comparison of this tensile strength with that of the members of the bicrystal and with that of the bulk tensile strength of granite.

At the inception of this research it was felt that suitable quartz-feldspar bicrystals could most easily be separated from pegmatites. Adhesive strength testing was initiated on bicrystals in the form of 11 mm (7/16 in) diameter bicrystals extracted from pegmatites from Custer County, South Dakota. The result of these tests (table 1) demonstrated the feasibility of adhesive strength testing and indicated that quartz-

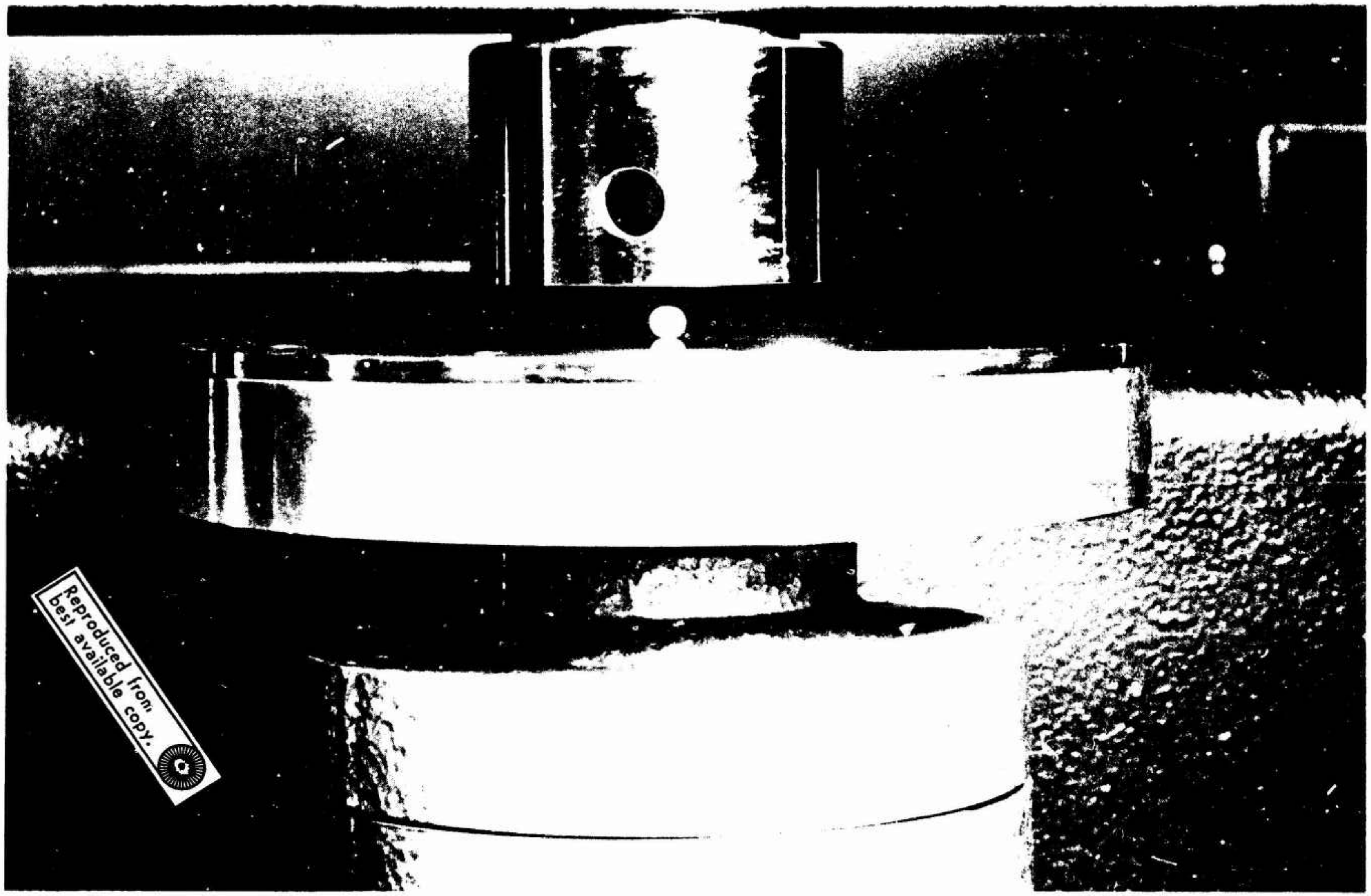

FIGURE 4. - Bicrystal in Place on the Load Cell of the Testing Machine.

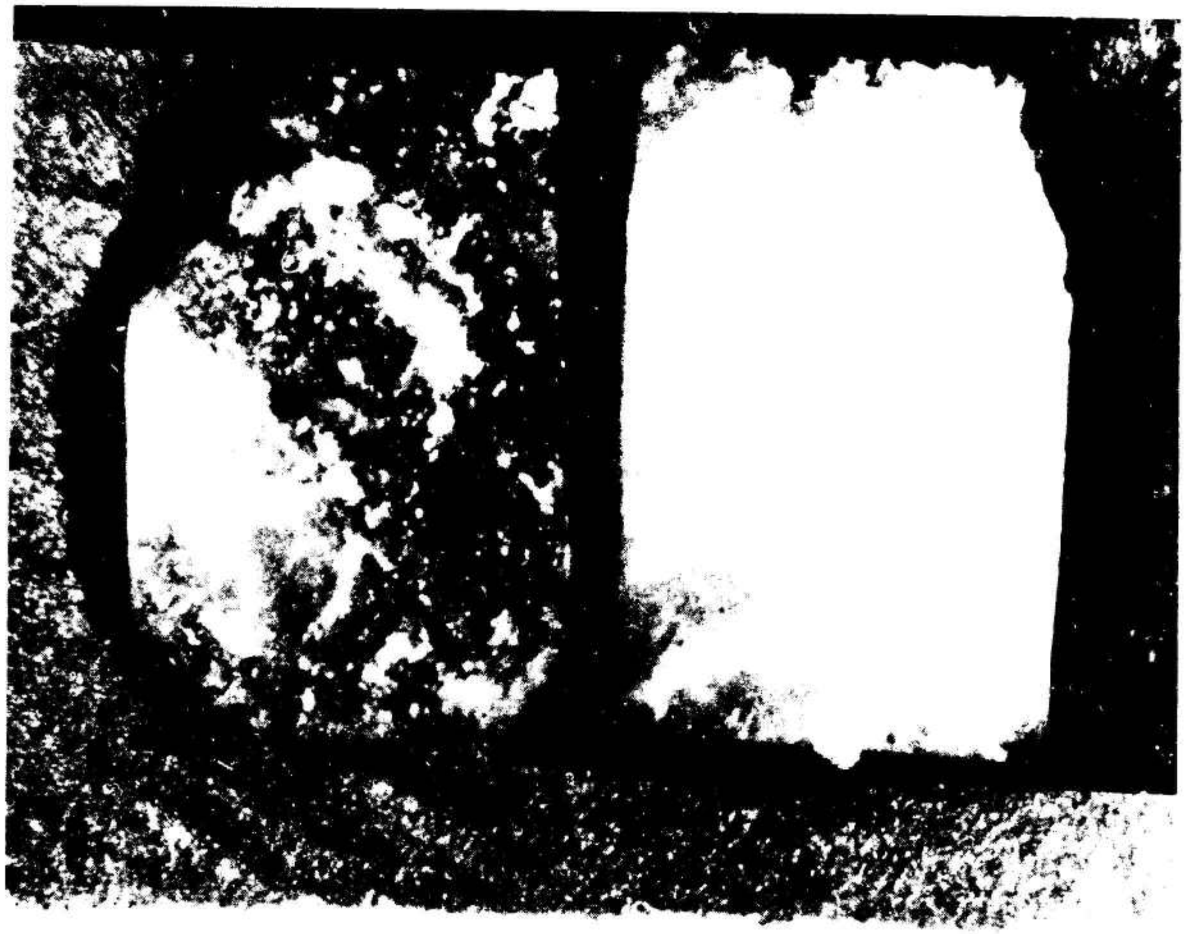

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FIGURE 5. - Bicrystal Broken at Quartz-Feldspar Interface.

TABLE 1. - Tensile strength of quartz-feldspar bicrystals separated from pegmatites

Quartz-feldspar bicrystal	Brazilian test TS (MN/m^2)	TS (psi)
X-1	5.31	770
X-2	5.48	795
X-3	3.87	561
X-4	4.87	784
X-6	3.82	554
X-9	4.66	676
X-13	3.84	558
X-14	5.88	852
X-17	5.51	799
X-18	9.13	1,324
X-21	5.23	758
X-23	4.29	623
X-26	8.92	1,294
X-27	4.04	587
X-28	9.35	1,355
X-20	8.64	1,252
Average	5.80	846

feldspar bicrystals remain strongly adherent after extraction from the rock.

After demonstrating the feasibility of adhesive strength testing, the drilling and tensile testing techniques were refined in order to study the intergranular adhesive strength of smaller bicrystals separated from finer grained rocks. Samples of the Rockville granite and some graphic granites from South Dakota and Connecticut were sliced into 3 mm (1/8 in) thick slabs. Small (5 mm diameter) quartz-feldspar bicrystals were extracted from these slabs using a 6 mm (1/4 in) O.D. diamond-tipped core drill. The extracted bicrystals were then subjected to the Brazilian test.

Table 2 is a compilation of selected-area tensile strength data for graphic granite from Custer County, South Dakota. These data indicate that the quartz and feldspar are strongly adherent (8.4 MN/m^2, 1,214 psi) at the crystalline interface but that their tensile strength is somewhat less than the average strength of the quartz and feldspar (12.5 and 13.3 MN/m^2, 1,810 and 1,933 psi respectively).

The ability to compare the tensile strength of the interface with that of the members of the bicrystal is essential because the geometrical restraints inherent in the research require the use of indirect tensile testing. This comparison provides a baseline with which to relate these measurements with direct tensile test data.

Graphic granite is especially amenable to this type of experimentation because it is coarse grained. The quartz and feldspar are intergrown in a geometrically regular pattern such that their crystalline interfaces are

TABLE 2. - Tensile strength of graphic granite (5 mm cores)

Bicrystal data			Single crystal data			Single crystal data		
Quartz-feldspar bicrystal	Brazilian test		Feldspar crystal	Brazilian test		Quartz crystal	Brazilian test	
	TS (psi)	TS (MN/m^2)		TS (psi)	TS (MN/m^2)		TS (psi)	TS (MN/m^2)
GG1	993	6.8	GGF1	1,429	9.8	GGQ1	1,850	12.8
GG2	805	5.5	GGF2	2,182	15.0	GGQ2	1,770	12.2
GG3	1,277	8.8	GGF3	2,228	15.4			
GG4	1,703	11.7	GGF4	3,688	25.4			
GG5	989	6.8	GGF5	2,322	16.0			
GG6	972	6.7	GGF6	857	5.9			
GG7	1,228	8.5	GGF7	1,642	11.3			
GG8	851	5.9	GGF8	2,483	17.1			
GG10	819	5.6	GGF9	1,729	11.9			
GG11	1,675	11.5	GGF10	2,604	18.0			
GG12	739	5.1	GGF11	788	5.4			
GG13	1,732	11.9	GGF12	1,212	8.4			
GG14	1,593	11.0	GGF13	3,121	21.5			
GG15	1,482	10.2	GGF14	1,041	7.2			
GG16	1,810	12.5	GGF15	2,601	18.0			
GG17	754	5.2	GGF16	2,082	14.4			
			GGF17	1,421	9.8			
			GGF18	1,454	10.0			
			GGF19	1,319	9.1			
			GGF20	2,462	17.0			
Averages	1,214	8.4	Averages	1,933	13.3	Averages	1,810	12.5

generally planar. The high degree of planarity of these interfaces permits a separation of crystals at the interface which is more exact than that exhibited by samples extracted from pegmatites or the Rockville granite.

Coarse grained pegmatites and graphic granites, the rocks used to establish the feasibility of adhesive strength testing, are not typical of the granitic rocks normally encountered in mining or tunneling. Thus, bicrystals separated from non-pegmatitic granites were included in order to make the results of this study more widely applicable.

Granites typically are composed of crystals from 0.1 to 2 mm in diameter. The procedure used in this study is not capable of extracting and testing bicrystals in this size range. Thus, only unusually coarse grained granites could be included in this study. The Rockville granite is the coarse grained granite chosen for study. It is quarried in the vicinity of St. Cloud, Minnesota, and is used as an architectural stone.

The tensile strength of 5 mm disks separated from selected areas in slabs of Rockville granite has been measured and tabulated (table 3). These data show that selected-area tensile strength measurements can be performed on granites and that quartz-feldspar interfaces are strongly adherent but have a lower tensile strength than the quartz and feldspar.

A comparison of the strength of quartz-feldspar interfaces with the bulk tensile strength of a series of granites can be made with reference to table 4. This table shows that the mean tensile strength of quart-feldspar interfaces separated from graphic granites and the Rockville granite is comparable with the mean tensile strength (measured by a Brazilian test) of a series of granites whereas bicrystals separated from coarse grained pegmatites are somewhat weaker.

TABLE 3. - Tensile strength of selected areas extracted from the Rockville Granite

SAMPLE CODE	Quartz-feldspar bicrystals that separated at inter-phase boundary		SAMPLE CODE	Feldspar		SAMPLE CODE	Quartz	
	Tensile strength			Tensile strength			Tensile strength	
	MN/m²	psi		MN/m²	psi		MN/m²	psi
1G	6.36	923	1F	16.06	2,330	1Q	13.84	2,008
2G	5.43	788	2F	16.64	2,414	2Q	16.03	2,325
3G	5.05	733	3F	7.34	1,064	3Q	10.79	1,565
4G	12.36	1,792	4F	28.97	4,201	4Q	24.69	3,581
5G	14.17	2,055	5F	18.46	2,677	5Q	9.83	1,426
6G	9.50	1,378	6F	13.60	1,972	6Q	15.52	2,252
7G	8.32	1,207	7F	12.43	1,803	7Q	13.73	1,991
8G	8.32	1,207	8F	10.56	1,532	8Q	10.18	1,477
9G	12.70	1,842	9F	16.61	2,409	9Q	5.68	824
10G	7.55	1,096	10F	21.97	3,186	10Q	12.20	1,769
11G	11.76	1,706	11F	10.48	1,572	11Q	12.17	1,765
12G	5.89	855	12F	11.67	1,693	12Q	15.53	2,253
13G	22.99	3,335	13F	17.39	2,522	13Q	15.60	2,263
14G	16.42	2,382	14F	22.74	3,297			
15G	7.80	1,131	15F	3.94	572			
16G	13.42	1,946	16F	13.44	1,950			
			17F	10.96	1,590			
Avg.	10.50	1,524	Avg.	14.90	2,164	Avg.	13.52	1,961

TABLE 4. - Tensile strengths of quartz-feldspar interfaces compared with the bulk tensile strengths of six granites

Bicrystal data[4]			Bulk granite data[5]		
Rock	Brazilian test		Brazilian test		Granite
	MN/m²	psi	MN/m²	psi	
Pegmatite	5.80	846	9.31	1,350	Warman
Graphic Granite	8.40	1,214	7.80	1,130	Lac Dubbonet
Rockville Granite	10.50	1,524	14.00	2,000	Rainbow
			10.00	1,400	Rockville
			15.00	2,200	Charcoal
			7.27	1,057	Barre

Microindentation hardness testing is included in this study of intergranular adhesion because it seems reasonable that a correlation should exist between hardness at grain boundaries and intergranular tensile strength. A method of selected area hardness testing has been developed to probe hardness variations at areas immediately adjacent to phase boundaries in rocks. A Tukon microindentation hardness tester was fitted with a Vickers indenter under a 50 gram load. The dimensions of ten indentations placed adjacent to the grain boundary are compared with ten indentations placed near the center of the grain. A computer program was written to compile the data and to perform a test of significance of the difference between the mean dimensions of the indentations in these areas.

Table 5 compares the microindentation hardness of quartz and feldspar near grain boundaries with the bulk hardness of these minerals in some igneous and metamorphic rocks. These hardness measurements were taken on dry polished sections and on some sections under various solvents.

[4] Average tensile strengths taken from tables 1, 2, and 3.
[5] Unpublished data from Property Determination Research Support Section, Twin Cities Mining Research Center.

Grain boundary hardening and softening was detected in quartzites and granites. The interpretation of these data is incomplete at present, but impurity segregation at the grain boundaries is thought to play a role in this phenomenon.

TABLE 5. - Comparison of the microindentation hardness of quartz and feldspar near grain boundaries with the bulk hardness of these minerals in some igneous and metamorphic rocks

Rock	Sample Number	Mineral	Environment	Hardness at grain boundary compared with bulk hardness		
				Harder	Softer	Same
Rockville granite	RGr-1A	Quartz	Dry			X
Do.	RGr-2B	do.	do.			X
Do.	RGr-2C	do.	do.			X
Do.	RGr-2	do.	Water	X		
Do.	RGr-2	do.	Glycerine (8%)		X	
Do.	RGr-3	do.	Water		X	
Do.	RGr-3	do.	Glycerine (8%)		X	
Do.	RGr-3	do.	DMF (50%)*			X
Do.	RGr-3	do.	DMSO (50%)**			X
Do.	RGr-3	Feldspar	DMF (50%)		X	
Do.	RGr-3	do.	DMSO (50%)			X
Do.	RGr-1C	do.	Dry		X	
Do.	RGr-2A	do.	do.			X
Do.	RGr-1D	do.	do.			X
Do.	RGr-1D	do.	do.			X
Do.	RGr-3A	do.	do.			X
Do.	RGr-2B	do.	do.			X
Do.	RGr-2C	do.	do.			X
Do.	RGr-2C	do.	do.			X
Do.	RGr-3	do.	Glycerine (8%)			X
Pegmatite	QF-1	Quartz	Dry			X
Do.	QF-3	Quartz	do.			X
Do.	QF-1	Feldspar	do.			X
Do.	QF-3	Feldspar	do.			X
Sioux quartzite	SQ-1A	Quartz	do.		X	
Do.	SQ-1A	do.	do.		X	
Do.	SQ-1C	do.	do.			X
Do.	SQ-1C	do.	do.		X	
Do.	SQ-1D	do.	do.			X
Do.	SQ-1D	do.	do.	X		
Wausau quartzite	Q-1	do.	do.	X		
Do.	Q-1	do.	do.		X	
Do.	Q-1	do.	Water	X		
Do.	Q-1	do.	do.		X	
Do.	Q-1	do.	do.			X
Do.	Q-1	do.	do.			X
Do.	Q-1	do.	do.			X
Do.	Q-1	do.	do.			X
Charcoal granite	ChGr-1A	do.	Dry		X	
Do.	ChGr-1B	Feldspar	do.			X
Do.	ChGr-1B	do.	do.			X
Labradorite	L-1A	do.	do.			X
Do.	L-1A	do.	do.		X	

*DMF = Dimethylformamide
**DMSO = Dimethylsulfoxide

ANALYSIS OF DATA AND CONCLUSIONS

Quartz-feldspar bicrystals can be selectively extracted from granitic rocks and broken in the immediate vicinity of the crystalline interface to obtain a measure of the strength of the intercrystalline bonding.

To the knowledge of the authors, the data presented above are the first direct measurements made of the strength of crystalline interfaces in rock. The strength of these interfaces is an important rock property. It can influence the path of cracks and hence can greatly influence the strength and mode of failure of rock.

This research has given some insight into the mechanism of intercrystalline bonding. The very fact that the crystals do not separate when they are extracted from the rock indicates that the crystals are bound together at their crystalline interface.

The experiments indicate that this bonding is fairly strong and the bicrystals appear to retain their adherency in thin section (i.e. when they are less than 2 mm thick). Thus the mechanism responsible for intergranular adhesion evidently operates on a microscopic scale.

A detailed atomistic picture of the mechanism responsible for this phenomenon is beyond the limits of our present knowledge, but it seems likely that the adherency is a result of chemical bonding between the surfaces of the quartz and feldspar, microscopic interfingering of the phases or a combination of both phenomena.

Attempts have been made to minimize the effect of strengthening the interfaces through interfingering of phases by selecting straight planar interfaces. It is doubtful, however, that this effect can be completely eliminated on a microscopic scale.

The strength of any chemically bonded area which may occur far exceeds the real strength of quartz and feldspar. Thus true atomic interfacial separation probably never occurs to any significant extent when mechanical forces are used to separate a pair of minerals that adhere because they have achieved atomic contact over all or any portion of an interfacial area. Thus it is not possible to interpret these data directly in terms of the strength or number of bonds at the crystalline interface.

Chemical bonds operate over very small distances. Hence two surfaces must be brought very close together for these forces to become operative. If the quartz and feldspar grains both had atomically smooth planar interfaces which were chemically bonded togehter, all attempts to separate them mechanically would result in the failure of the bulk of either quartz or feldspar.

Crystalline interfaces in rock differ from this idealization because they are rough and contaminated. These imperfections contribute to a greatly decreased real area of contact. Thus when a quartz-feldspar interface which has locally achieved real contact is separated mechanically, a little of the quartz remains on the feldspar surface and vice versa.

The proportion of the fracture surface area covered by remnants may be a rough measure of the spatial extent of bonding across the interface.

Figure 6 shows a bicrystal which was bonded over a large portion of the crystalline interface, whereas figure 7 shows large smooth areas which evidently were not bonded.

The most significant outcome of this research is the demonstration that small scale selected area tensile strength testing is feasible in rocks. This technique can be applied not only to intergranular adhesive strength testing but also to the determination of the strength at any selected region within the rock and hence is potentially useful in rock fragmentation research.

FIGURE 6. - Quartz-Feldspar Crystalline Interface With Small Proportion of Unbonded Area.

FIGURE 7. - Quartz-Feldspar Crystalline Interface With a Small Proportion of Bonded Area.

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